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(71) Applicant  
**Lion Corporation (Japan),**  
**3-7 Honjo 1-chome, Sumida-ku, Tokyo-to, Japan**

(72) Inventors  
**Takako Hama,**  
**Futao Wakui**

(74) Agent and/or Address for Service  
**Boult, Wade & Tennant,**  
**27 Funnival Street, London EC4A 1PD**

(54) **Shampoo compositions**

(57) A shampoo composition which is non-irritative to the skin comprises, beside a detergent surface active agent, up to 30% by weight of an extract from the barks or wooden xylems of white birch, e.g. *Betula alba*, and up to 20% by weight of a sugar alcohol ester or a fatty acid ester of polyglycerin, the balance being water and additives.

GB 2 140 452 A

## SPECIFICATION

## Shampoo compositions

5 The present invention relates to a novel shampoo composition which causes little irritation to the skin and is tender to the hair despite a good detergent power and excellent in the pleasant touch of the lather and in the hair conditioning power imparting smoothness to the hair shampooed therewith. 5

Most conventional shampoo compositions are formulated with a surface active agent such as sodium alkylbenzene sulphonates, salts of higher alcohol sulphate esters and salts of polyoxyethylene alkylsulphates as the principal ingredient. Such a detergent component is of course irritant to the skin so it has been proposed to formulate shampoo compositions with a hydrolyzate of proteins, hydrolyzate of acylated proteins, amino acids and the like with the object of mitigating the irritation to the skin caused by the shampoo composition. The addition of these additives to a shampoo composition is, however, not sufficiently effective to reduce the irritation caused by the surface active agent. It is taught in Japanese Patent Publication 46-19635 that a considerable mitigation of irritation can be obtained when a detergent composition containing a sodium alkylbenzene sulphonate or a salt of a polyoxyethylene alkylphenyl ether sulphate ester as the principal surface active agent is admixed with saponin and aspartic acid or sucrose in combination. When these additives are added to a shampoo composition in combination, however, such a shampoo composition is not always acceptable in respect of the smoothness of the hair shampooed therewith although the irritation to the skin is mitigated. 10 15 20

In view of the above described state of the art, the inventors have continued extensive investigations on shampoo compositions and arrived at a discovery that the irritativeness of a conventional shampoo composition containing a surface active agent can be greatly mitigated while a pleasant touch of the lather and smoothness of the hair shampooed therewith is obtained and the detergent power and lathering thereof are not affected when the shampoo composition is admixed with an extract from bark or wooden xylem of white birch together with a sugar alcohol ester or a fatty acid ester of polyglycerin. 25

Thus, the shampoo composition of the present invention comprises, in addition to a detergent surface active agent, greater than 0.0005% by weight of an extract from the bark and/or wooden xylem of white birch belonging to the genus *Betulaceae* and greater than 0.1 % by weight of a sugar alcohol ester or a fatty acid ester of polyglycerin, the balance being water and optional additives, e.g. as conventional in shampoo compositions. 30

The invention includes a method of washing hair which comprises using a shampoo of the invention. Such a method may, of course, be applied commercially in the hairdressing industry.

The detergent surface active agent as the principal ingredient in the inventive shampoo composition may be any one of the conventional surface active agents used in detergent compositions without particular limitation, including anionic, cationic, non-ionic and amphoteric ones. The amount of the surface active agent in the inventive shampoo composition should usually be in the range from 3 to 30% by weight. 35

Exemplary of the surface active agents suitably formulated in the inventive shampoo composition are anionic surface active agents, for example salts of alkyl benzene sulphonic acids, salts of polyoxyethylene sulphates, sulphate esters of higher alcohols, salts of alkylnaphthalene sulphonic acids, salts of alkylphosphates and salts of  $\alpha$ -olefin sulphonic acids; non-ionic surface active agents, for example polyoxyethylene alkyl ethers, polyoxyethylene alkyl phenyl ethers, sorbitan fatty acid esters and polyoxyethylene acyl esters; cationic surface active agents, for example quaternary ammonium salts; and amphoteric surface active agents such as alkyl betaines, for example. 40

The characteristic ingredient in the inventive shampoo composition is an extract from bark and/or wooden xylem of white birch, which is a deciduous arborescent tree belonging to the genus of *Betulaceae* including *Betula tauschii* and *Betula alba*. Extracts from the bark or wooden xylem of white birch contain large amounts of tannin, saponin and betulin, lupane-like terpenes and, in addition, contain essential oils, bitters and betulosid in small amounts. In particular, the contents of betulin and tannin are high in the bark sometimes reaching 10% by weight or higher and up to 15% by weight, respectively. 45 50

The extract from the bark and/or wooden xylem of white birch is prepared, for example, by dipping minced and air-dried bark and/or xylem of white birch in a suitable volume of a solvent mixture of water and ethyl alcohol for 1 to 2 weeks followed by the removal of the solid matter by a suitable method such as centrifugal separation to give an extract solution which is kept for several days in a dark and cool place and then filtered to remove precipitates, if any. 55

The amount of the white birch extract in the inventive shampoo composition is, calculated as the dried material after heating at 105°C for 6 hours, greater than 0.0005 by weight and preferably not more than 30% by weight of the composition or, more preferably, from 0.05 to 10% by weight of the composition. The desired effects to be obtained by the addition of the white birch extract cannot be obtained with an amount thereof smaller than above while an adverse effect of decreasing the clarity of the shampoo composition is caused with the lapse of time by the addition of the extract in excess of 30% by weight. 60

Another essential and characteristic ingredient in the inventive shampoo composition is, in combination with the above described extract of the bark and/or xylem of white birch, a sugar alcohol ester or a fatty acid ester of polyglycerin.

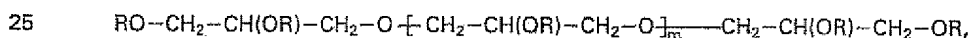
The sugar alcohol ester is a compound typically represented by the general formula  $R^1(-O-CO-R^2)_n$ , in 65

which R<sup>1</sup> is a saccharide residue, R<sup>2</sup> is an alkyl group having from 10 to 18 carbon atoms and n is an integer of 1 or 2 for a monoester and diester, respectively. Mixtures of a mono- and diesters are of course suitable. When the number of the carbon atoms in the alkyl group denoted by R<sup>2</sup> is 9 or smaller, the lather formed of the shampoo composition formulated with the sugar alcohol ester has a less pleasant touch while a sugar alcohol ester in which the alkyl group has 19 or more carbon atoms is undesirable due to the lower dispersibility in the shampoo composition. The saccharide from which the saccharide residue denoted by R<sup>1</sup> is derived may be saccharose, maltose or lactose though not particularly limited thereto.

The sugar alcohol ester used in the inventive shampoo composition is usually prepared in the following manner. A solution of saccharose or other saccharide in dimethyl formamide is admixed with a fatty acid methyl ester to form an apparently clear microemulsion and heated under reduced pressure in the presence of an alkali catalyst to effect the ester exchange reaction. Alternatively, the ester exchange reaction is performed in a clear emulsified state of the solution of the saccharide in water or in propylene glycol in which the fatty acid ester is dispersed in fine particles or droplets in the presence of a fatty acid soap.

The amount of the sugar alcohol ester in the inventive shampoo composition should be above 0.1% by weight and desirably in the range from 0.1 to 20% by weight or, preferably, from 1 to 10% by weight based on the composition. The desired effects to be obtained by the addition of the sugar alcohol ester are not shown when the amount thereof is too small while too large an amount of the sugar alcohol ester is undesirable due to the possible degradation of the composition in time in respect of the colour change from light yellow to deep brown.

An ingredient which is an alternative to the above described sugar alcohol ester is a fatty acid ester of polyglycerin, which may be prepared by the esterification reaction of a fatty acid ester on a polyglycerin which is a dehydration condensation product of glycerin. The compound is typically represented by the general formula



in which each R is a hydrogen atom or a fatty acid residue independently from the others, at least one of the Rs in a molecule being a fatty acid residue, and the suffix m is zero or a positive integer. The fatty acid from which the fatty acid residue denoted by R is derived may be a saturated or unsaturated fatty acid having a linear or branched molecular structure. The fatty acid should preferably have from 10 to 20 or, more preferably, from 12 to 18 carbon atoms in a molecule. Exemplary of such a fatty acid are lauric, myristic, palmitic, stearic, isostearic and oleic acids. When the number m is equal to zero, the ester is a diglycerin ester and the value of m, when it is a positive integer, should typically not be larger than 30 or, preferably, than 20. A higher polyglycerin ester in which the number m is larger than 30 is undesirable due to the tendency of hair shampooed by use of a shampoo composition formulated therewith to have a sticky touch. Fatty acid esters of glycerin are not suitable in the inventive shampoo composition in respect of the touch of the lather and the smoothness of the hair shampooed by use of a shampoo composition formulated therewith.

Amongst the preferred fatty acid esters of polyglycerin are laurate, myristate, palmitate, oleate, pentastearate, decastearate and decalostearate of decaglycerin, stearate, oleate and hexaoleate of hexaglycerin and diglycerin stearate.

The amount of the fatty acid ester of polyglycerin in the inventive shampoo composition should be greater than 0.1% by weight and desirably in the range from 0.1 to 20% by weight or, preferably, from 0.1 to 10% by weight based on the composition although the amount thereof should be determined in consideration of the amount of the white birch extract formulated in the composition.

The inventive shampoo composition is readily prepared by dissolving the above described components in water each in the specified amount. It is of course optional that the inventive shampoo composition is further admixed with various additives, e.g. as conventionally added to shampoo compositions, including, for example, organic and inorganic builders, carboxymethylated cellulose, lower alcohols, fluorescent brightening agents, colouring materials or perfumes.

Following are the examples to illustrate the formulation and advantages of the inventive shampoo composition in more detail making comparison with some comparative compositions. In the examples, testing and evaluation of each shampoo composition were undertaken in the following manner.

#### (1) Organoleptic evaluation of irritativeness to skin

The shampoo composition under testing was diluted 100 times with water and 10 tester members each had their hand dipped in the diluted solution at 35 °C for 1 minute followed by drying for 1 minute in dry air repeating this cycle of dipping and drying 15 times. After 24 hours from this 15 times repeated cycles of dipping in the solution and drying, the condition of skin chapping was visually examined and graded in A, B and C according to the following criteria.

- A: Almost no chapping was found on the skin.
- B: Symptom of xerodermic desquamation was found on a part of the strata cornea of the skin.
- C: Symptom of xerodermic desquamation was found in 3 members or more of the testers.

(2) *Irritateness to skin in correlation to BSA molecular ellipticity*

The irritateness of the shampoo composition to skin was correlated to the molecular ellipticity ( $\theta$ ) of bovine serum albumin (BSA molecular ellipticity) in the solution under testing as an indicative parameter in the following manner.

- 5 Thus, the 1%-diluted shampoo solution under testing was admixed with 100 ppm of bovine serum albumin and further with a sodium phosphate buffer solution to give a concentration of 50 mM and the circular dichroism thereof was determined at 25 °C in a JASCOCD Spectropolarimeter from which the value of the molecular ellipticity was calculated and compared with the molecular ellipticity of an unmodified bovine serum albumin which was  $-11.3 \times 10^6 \text{ deg}\cdot\text{cm}^2/\text{decimole}$ . It is known that a test solution in which the BSA molecular ellipticity is the same as or close to that of the unmodified bovine serum albumin as a measure of the absence of denaturation of the bovine serum albumin is little irritative to skin.

(3) *Lathering power*

- 15 A 20 ml portion of a 6% aqueous solution of the shampoo composition was taken in a 100 ml graduated cylinder with admixture of 0.2 g of liquid lanolin as a simulated filth and the mixture was shaken at 25 °C 20 times within 10 seconds to record the volume of the lather in ml after 1 minute of standing.

(4) *Organoleptic evaluation of lather touch*

- 20 The touch of the lather formed of the shampoo composition was organoleptically evaluated in comparison with a sodium polyoxyethylene alkyl sulphate (AES-Na) as a reference shampoo by 10 tester members. The hair of each of the tester members was parted in the middle to right and left portions and lather was formed in each portion by stirring 3 g of the reference shampoo or a shampoo under testing to make comparison for the touch of the lather between the right and left portions. The AES-Na as the reference shampoo had an average number of moles of ethylene oxide addition  $\bar{P}$  of 3 and the molar ratio of the  $C_{12}$  alkyl to the  $C_{13}$  alkyl groups was 1/1.

The results of the above described organoleptic test were summarized for the 10 members and graded into 5 according to the following criteria.

- 30 ++: apparently more pleasant lather touch than AES-Na  
 +: somewhat better lather touch than AES-Na  
 ±: about the same lather touch as AES-Na  
 -: less pleasant lather touch than AES-Na  
 --: much poorer lather touch than AES-Na

35 (5) *Smoothness of shampooed hair*

- A 20 cm long hair bundle weighing 5 g was shampooed using the shampoo composition under testing followed by rinse and drying and the smoothness thereof was evaluated in the one-pair comparison method by 20 tester members with that of a hair bundle shampooed similarly by use of a 20% aqueous solution of sodium dodecylsulphate (SDS) as the reference. The results were graded in A, B and C according to the following criteria.

- 45 A: apparently better smoothness than SDS  
 B: somewhat better smoothness than SDS  
 C: about the same smoothness as SDS

*Examples and comparative examples*

Table 1 below gives the formulations of 13 shampoo compositions according to the invention and 9 comparative shampoo compositions. The ingredients numbered I to XI, of which the contents in the composition are given in % by weight in the table, the balance being water, are as follows.

- 50 I: sodium polyoxyethylene alkyl ether sulphate  
 II: sodium salt of coconut oil alcohol sulphuric acid ester having a molecular weight of 305  
 III: triethanolamine salt of coconut oil alcohol sulphuric acid ester having a molecular weight of 305  
 55 IV: a sulphonation product of a  $C_{14}$ - $\alpha$ -olefin with sulphuric anhydride followed by neutralization and hydrolysis with sodium hydroxide having a molecular weight of 308  
 V: triethanolamine acylglutamate  
 VI: extract from white birch (*Betula alba*) bark added as a 50% ethyl alcohol solution (amount calculated as dried at 105 °C for 6 hours)  
 VII: sugar ester with lauric acid, monoester/diester = 7/3  
 60 VIII: monoester of lactitol with lauric acid  
 IX: monoester of maltitol with stearic acid  
 X: decaglycerin stearate  
 XI: decaglycerin oleate

The results of the evaluation of each shampoo composition for 5 items are summarized in Table 2 below. As is clear from these results, the comparative shampoo compositions were not satisfactory in one or more of the evaluated items due to the omission of either one or both of the essential ingredients including the white birch extract and the sugar alcohol ester or the fatty acid ester of polyglycerin.

5

5

TABLE 1

Ingredient											
	I	II	III	IV	V	VI	VII	VIII	IX	X	XI
Comparative Example	1	20									
	2		20								
	3			20							
	4				20						
	5					20					
	6	15					5				
	7		15				5				
	8			15			5				
	9				15					5	
Inventive Example	1		15			5	5				
	2		15			5	5				
	3			15		5	5				
	4				15	5	5				
	5	15				5		5			
	6	15				5			5		
	7	15				0.0005	0.1				
	8	15				30.0	20				
	9				15	5				5	
	10				15	5					5
	11	15				5					
	12				15	0.0005				0.1	
	13				15	30.0				20	
	14*	15				5	5				
	15*	15				5				5	

\*) The component VI was an extract from wooden xylem of white birch.

TABLE 2

			<i>Irritative- ness to skin (orga- noleptic)</i>	<i>BSA molecu- lar ellip- ticity (*1)</i>	<i>Lathering power, cm<sup>3</sup></i>	<i>Touch of lather</i>	<i>Smooth- ness of shampooed hair</i>	
5								5
		1	C	- 9.3	50	±	C	
		2	C	- 9.0	80	++	C	
10	Comparative Example	3	C	- 9.0	80	++	C	10
		4	C	- 8.7	70	±	C	
		5	A	-11.1	60	-	B	
		6	A	-10.9	60	±	B	
		7	A	-10.8	80	++	C	
15		8	A	-10.6	70	±	C	15
		9	A	-11.2	65	±	B	
		1	A	-11.3	70	++	A	
		2	A	-11.3	70	++	A	
20		3	A	-11.3	70	++	A	
		4	A	-11.3	70	++	A	20
		5	A	-11.3	70	++	A	
		6	A	-11.3	70	++	A	
	Inventive Example	7	A	-11.3	70	++	A	
		8	A	-11.3	70	++	A	
25		9	A	-11.3	70	++	A	25
		10	A	-11.3	70	++	A	
		11	A	-11.3	70	++	A	
		12	A	-11.3	70	++	A	
		13	A	-11.3	70	++	A	
30		14	A	-11.3	70	++	A	30
		15	A	-11.3	70	++	A	

(\*1) Unit:  $\times 10^{-6}$  deg·cm<sup>2</sup>/decimole

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## CLAIMS

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1. A shampoo composition which comprises:
  - (a) a detergent surface active agent;
  - 40 (b) greater than 0.0005 by weight, calculated on a dry basis, of an extract from the bark or wooden xylem of white birch belonging to the genus of Betulaceae; and
  - (c) greater than 0.1 by weight of a sugar alcohol alkyl ester or a fatty acid ester of polyglycerin; the balance being water and optional additives.
2. A shampoo composition according to Claim 1 wherein component (a) is in an amount of from 3 to 30% by weight.
3. A shampoo composition according to Claim 1 or Claim 2 wherein component (b) is in an amount of not more than 30% by weight calculated on a dry basis.
4. A shampoo composition according to any one of the preceding claims wherein component (c) is in an amount of no greater than 20% by weight.
- 50 5. A shampoo composition according to any one of the preceding claims wherein the white birch is *Betula alba*.
6. A shampoo composition according to any one of the preceding claims wherein the sugar from which the sugar alcohol alkyl ester is derived is saccharose, maltose or lactose.
7. A shampoo composition according to any one of the preceding claims wherein the alkyl group
- 55 forming the sugar alcohol alkyl ester has from 10 to 18 carbon atoms.
8. A shampoo composition according to any one of the preceding claims wherein the sugar alcohol alkyl ester is a monoester or diester of a sugar with alkyl groups.
9. A shampoo composition according to any one of Claims 1 to 5 wherein the degree of polymerization of the polyglycerin is in the range from 2 to 30.
- 60 10. A shampoo composition according to any one of Claims 1 to 5 or 9 wherein the fatty acid forming the fatty acid ester of polyglycerin with the polyglycerin has from 10 to 20 carbon atoms in a molecule.
11. A shampoo composition according to any one of Claims 1 to 5, 9 or 10 wherein the fatty acid ester of polyglycerin is laurate, myristate, palmitate, oleate, pentastearate, decastearate or decalostearate of decaglycerin, stearate, oleate or hexaoleate of hexaglycerin, or diglycerin stearate.
- 65 12. A shampoo composition substantially as hereinbefore described in any one of Examples 1 to 15.

13. A method of washing hair which comprises using a shampoo composition according to any one of the preceding claims.

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